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ANALYSIS OF LIQUID ROCKET TANKAGE FOR MODEL LR58-RM-4 LIQUID PROPELLANT THRUST UNIT FOR THE BULLPUP MISSILE

E. J. King, et al

Bell Aerospace Company

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ANALYSIS OF LIQUID ROCKET TANKAGE FOR MODEL LR58-RM-4 LIQUID PROPELLANT THRUST UNIT FOR THE BULLPUP MISSILE

FINAL REPORT

Bell Aerospace Company
Division of Textron, Inc.
P.O. Box One
Buffalo, New York 14240

Authors. E. J. King
J. Salvaggi

FEBRUARY 1976

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AIR FORCE ROCKET PROPULSION LABORATORY
DIRECTOR OF SCIENCE AND TECHNOLOGY
AIR FORCE SYSTEMS COMMAND
EDWARDS, CALIFORNIA 93523

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FOREWARD

This report was submitted by the Bell Aerospace Company, Division of Textron, Inc., P.O. Box One, Buffalo, New York 14240, under Contract No. F04611-74-C-0007, Supplemental Agreement P0000[illegible], Job Order No. 305811 TR with the Air Force Rocket Propulsion Laboratory, Edwards, CA 93523.

The Project Manager was E. J. King; the Project Metallurgical Engineer was John Salvaggi. The in-depth analyses were conducted by John Salvaggi. Analysis of corrosion products was performed by D. G. Roberts and Peter Yin.

This report has been reviewed by the Information Office/DOZ and is releasable to the National Technical Information Service (NTIS). At NTIS it will be available to the general public, including foreign nations.

This report is unclassified and suitable for general public release.

Oree Dyes

OREE DYES, GS-13
Project Engineer

Clark W. Hawk, CS-14
Chief, Propulsion Applications Branch

FOR THE COMMANDER

Charles E. Sieber

CHARLES E. SIEBER, Lt/Col., Chief
Liquid Rocket Division

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The objective of this program was the assessment of Bullpup missile tankage and component compatibility with IRFNA and MAF-1 propellants, for a period of 10 - 12 years. Tankage material was the 2014-T6 aluminum alloy.

Evaluation of the tank and component surfaces disclosed only minor corrosion effects of a general, surface nature.

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The degree of material degradation which occurred is considered insignificant and would not alter the functional capability of the missile.

PREFACE

This report covers the examination and metallurgical evaluation of a liquid propellant thrust unit tankage assembly, Model No. LR58-RM-4, from a Bullpup missile. The primary purpose of this effort was to determine the presence of any damage sustained by the tankage and related components, after a long term exposure of 10 - 12 years to IRFNA and MAF-1 propellants.

The results of this long term exposure indicated negligible corrosion or damage of the aluminum alloys used in fabricating the subject unit. The degree of corrosion which did occur is considered insignificant and would not affect the functional capability of the missile system. Some slight corrosion of stainless steel components present in the oxidizer tank was also noted but should present no serious problems.

A high order of compatibility between the aluminum alloys of construction and the propellants stored in the thrust unit was demonstrated for the 10 - 12 year storage period involved.

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SECTION I

INTRODUCTION

The defueling of Bullpup missile tanks that have been stored for time periods of 10 - 12 years with MAF-1 fuel and Type III-A IRFNA oxidizer has presented an excellent opportunity to evaluate the corrosion behavior of actual flight hardware after extended, long term propellant storage. The subject program, which is a supplement to the original contract, initiated in October, 1973, involved a systematic study and analysis designed to determine the condition of two Bullpup missile tanks, one for fuel and one for oxidizer, together with associated components. The following procedure was used for this analysis:

1. Documentation of the as-received condition of Bullpup missile tankage.
2. Definition of anomalies and defects that altered the functional capability of missile tankage and components.
3. Submittal of metallurgical analysis report for PCO approval.
4. In-depth metallurgical analysis of anomalies or defects defined above.
5. Preparation of final summary report.

The evaluation program was divided into two phases. Phase I included steps 1 through 3 above, while Phase II (steps 4 and 5) dealt with in-depth and confirmation analyses of areas displaying visible evidence of corrosion. Metallurgical examination of the exposure vessels and associated components identified the nature and extent of corrosion that had occurred over the 10-year storage period. Anomalies were related primarily to exposure conditions. Processing and environmental effects in the post-storage period were considered in analyzing observed anomalies or defects.

Mechanical properties of specimens machined from the tank shell walls and selected components were also determined. These tests were considered necessary to verify heat treat condition and to establish the extent of any degradation which may have been caused by the long term exposure.

The ultimate purpose of this overall effort was to establish the compatibility characteristics of various tankage/component materials with earth storable liquid rocket propellants, over extended, long term storage periods.

Documentation of all areas of interest, including tank shell exterior and interior surfaces, weld geometry, corrosion and microstructure was performed throughout the program to insure completeness of the overall investigation.

SECTION II

PROGRAM STRUCTURE

This contract extension effort differed from the initial study, which involved the evaluation of thin wall propellant storage vessels. Oxidizer and fuel propellants were also different. However, the technical approach used was identical.

The storage tanks evaluated in the current program were of a heavy wall construction, integral with the missile and aligned in tandem, with welds joining each tank to a center bulkhead forging. Ancillary components contained within the tanks were also exposed to the propellants. The internal environments were as follows:

FUEL - Mixed Amine MAF-1

Diethylene Triamine (DETA)	- 50.5% by weight
Unsymmetrical Dimethylhydrazine (UDMH)	- 40.5% by weight
Acetonitrile (RMD Specification 4034)	- 9.0% by weight

OXIDIZER - IRFNA, Type III-A per MIL-N-7254C

No leakage of the missile tanks occurred in the 10-year storage period. Effort was, therefore, concentrated on examining surface effects produced by propellant contact with the aluminum alloy tank structure, aluminum alloy components and weldments and several stainless steel components contained within the tank shells.

SECTION III

TEST PROCEDURES

A. POST STORAGE TANKAGE ANALYSIS

The test hardware examined during this program is identified as Model No. LR58-RM-4 packaged liquid propellant thrust unit for the Bullpup B missile, Serial Number 19918. It was manufactured by the Thiokol Chemical Corporation, Reaction Motors Division. Extensive qualification testing performed prior to acceptance by the U.S. Bureau of Naval Weapons, verified the structural integrity of the unit.

The metallurgical procedure used in assessing corrosion damage involved a thorough examination of external and internal surfaces of the integral, propellant containing tank sections. Associated components contained within the tank sections were also subjected to a detailed examination. Documentation of all significant observations was performed to provide a data base from which subsequent in-depth and confirmatory analyses could be selected, thus fulfilling the requirements of the subject contract.

Sectioning of areas of interest, for a more detailed metallographic study was also performed. This approach permitted a more thorough analysis of the extent of any corrosion which had occurred, evaluation of weld geometry and quality and characterization of microstructure.

B. DETAILED ANALYSIS PROCEDURE

Those anomalies selected for additional, detailed analysis, as dictated by contract requirements, followed a format similar to that established for the original contract effort completed in December 1974 (Reference 1).

The procedure used, which was submitted to the project control officer for approval prior to initiation of the analyses, is described in the following sub-section.

1. Appearance Documentation

   (a) Cut anomaly and surrounding area from tank or component for ease of handling.

   (b) Take photomacrographs of anomaly surface; remove any corrosion products or deposits for analysis; take additional photomacrographs if any change in surface appearance is noted.

   (c) Expose any hidden surfaces by sectioning away from the externally corroded area. Take photomacrographs of any corrosion present.

2. Microstructure and Relation to Corrosion or Other Anomaly

   (a) Mount a cross section of critical area of anomaly.

   (b) Polish using conventional metallographic techniques.

(c) Examine in unetched condition for corrosion penetration of grain boundaries or similar effects and take appropriate photomicrographs.

(d) Etch with appropriate reagents to develop microstructure of weld and/or parent metal.

(e) Examine and take photomicrographs of microstructure, both as it relates to corrosion effects and also to determine matrix microstructure and material effects.

3. Chemical Analysis of Corrosion Products, Residual Deposits and Corroded Material

   (a) If corrosion products or residual deposits were removed in Step 2(a), analyze by infrared, X-ray diffraction or other appropriate analysis techniques.

   (b) If any suspicion exists that tank or component materials are not of the alloy expected (based on microstructure or other observations) spectrographic analysis will be performed.

C. CONFIRMATORY ANALYSIS

The related anomaly selected for confirmatory analysis will be evaluated in less detail than previously described for the detailed analysis. The procedure to be used is as follows:

1. Anomalies in other components which appear to be closely related to the subject selected for detailed analysis will have been identified.
2. The selected anomaly will be photographed to show surface appearance to the extent necessary to establish similarities to the detailed analysis subject.
3. The anomaly will then be sectioned, mounted and metallographically polished. It will be examined and photographed in both unetched and etched conditions, in the same manner as detailed analysis Steps 2(c) through 2(e).

D. METALLURGICAL ANALYSIS AND PREPARATION OF REPORT

The mechanical properties of the oxidizer and fuel tank shells will be established. These properties and the foregoing metallographic examination results will be reviewed and correlated with prior fabrication and storage history of the missile.

A final metallurgical analysis report will be prepared. The report will include glossy print reproductions of all applicable photographs showing surface appearance, corrosion products and microstructure. The individual sections of the text that will be included for each analysis are outlined below.

1. Test History

Brief statement summarizing unit identification, material of construction, propellant involved and leak or other anomaly description.

2. Observations

The text and related figures, primarily photomacrographs and photomicrographs, which describe and document all metallurgical observations made on the anomaly and surrounding material. Included in this section will be details of exposure, as available from AFRPL records and any other supporting information which is available.

3. Metallurgical Analysis

This section will summarize and discuss the above observations. Contrasts and similarities with anomalies in other tanks of this program, or perhaps with other similar hardware known to BAC metallurgists will be pointed out. The significance of the observations will be discussed and those observations of greatest importance to the analysis will be highlighted.

4. Confirmatory Analysis

Related anomaly, evaluated in sufficient depth to establish similarity to the detailed analysis subject, will be discussed briefly in this section.

SECTION IV

FABRICATION HISTORY OF THRUST UNIT

In the analysis of corrosion behavior of a metallic component or structure, it is instructive and often necessary to know the fabrication procedures and processing details involved, in order to reach logical conclusions as to the cause and significance of observed corrosion effects. The fabrication history, summarized briefly in this section, was obtained from information contained in the Reference 2 Qualification Report submitted to the U.S. Naval Bureau of Weapons by the Reaction Motors Division of the Thiokol Chemical Corporation. Specific details of the fabrication process were not covered in the referenced report; only a general overview of materials used and a functional description of internal components was presented.

The fuel and oxidizer tanks are in tandem, integral with the missile body. Both tanks consist of approximately 1/4-inch thick cylindrical sections, machined from 2014-T6 aluminum alloy forgings. They are welded to a central header or bulkhead, also machined from a 2014-T6 forging. Pressurizing inlet passages to each propellant tank are sealed by welded burst bands, probably fabricated of a low strength aluminum alloy or commercially pure aluminum. These are designed to rupture under gas generator pressure during the initial phase of ignition. Gas diffusers in each tank, located just aft of the burst bands, are fabricated of Type

321 stainless steel. A conical shaped aluminum alloy shock baffle is located in the oxidizer tank and formed to fit around the steel lined pressurization passages machined into the center header forging. The vortex director baffle is welded to the gas generator tube in the oxidizer tank. Both of these components appear to be of the same aluminum alloy composition.

The thrust chamber, situated on the central axis of the fuel tank, is a tubular element regeneratively cooled by fuel propellant flowing through a corrugated baffle surrounding the thrust chamber. Both of these components are fabricated of aluminum alloys. Stainless steel screens are placed in both tanks to filter the propellant flow upstream of the liquid injection orifices.

The precise sequence of operations or detailed fabrication procedures used to manufacture the Bullpup missile thrust unit was not available. However, general knowledge regarding aluminum alloy properties, preferred fabrication and welding procedures used in the aerospace industry and corrosion resistance in various media enabled a technically sound evaluation of the observed anomalies.

These elements and components of interest were in contact with the fuel or oxidizer liquid during the full storage period of the thrust unit. The effect of long term propellant exposure on the integrity of these components, changes in surface appearance of the wrought material and welded joints as a result of chemical reactions and the analysis of corrosion products or propellant residues comprised the major effort expended on this program.

SECTION V

DISCUSSION OF RESULTS

A. MACROSCOPIC EXAMINATION OF EXTERNAL AND PROPELLANT EXPOSED TANK SHELL SURFACES

1. External Surface

The external surfaces of the missile thrust unit were painted. The paint coating remained complete and protective, with virtually no chipping or peeling. No evidence of surface deterioration or corrosion was found, indicating no leakage of propellants and a relatively mild storage area environment. A view of the as-received thrust unit is presented in Figure 1.

2. Internal Surface

The entire unit was sectioned lengthwise, at the approximate center line of construction. A heavy duty band saw was used for this purpose. An overall view of the internal surfaces is presented in Figure 2.

A vastly different appearance was observed between the fuel and oxidizer tanks. Scattered, rust-like deposits and considerable loose residue were present on the internal surface of the oxidizer tank. The major portion of the surface was only dulled, with a discontinuous white to light green, powdery deposit evident. A metallic luster remained in evidence. Water line markings were also visible, indicating some residual liquid had remained in the tank, then evaporated.

Some areas of the tank contained a two layer deposit. The outer layer was a tan, scale like deposit, brittle and easily flaked off. The inner layer was a dark green in color, more adherent and less brittle. Both layers were easily removed, indicating that they were possibly residues remaining after evaporation of the propellants, and/or rinse solutions. Photomacrographs of this two layer deposit are shown in Figure 3. The metal surface beneath these deposits displayed a shallow, general corrosion pattern at magnifications to 60X. An in-depth analysis of this anomaly is presented in Section V (D) 2.

The surface of the fuel tank which contained MAF-1, a mixed amine, was discolored or etched to a gray-black color. Unlike the oxidizer tank, there was no loose residue present, only a tightly adherent film, indicating a surface reaction with the fuel had probably occurred. Scraping a small area of the film disclosed it to be a white, crystalline appearing substance. The surface beneath this film displayed negligible corrosion at magnifications to 60X. A further, in-depth analysis of this surface anomaly was performed and is presented in Section V (D) 3.

B. GENERAL EXAMINATION OF PROPELLANT EXPOSED COMPONENTS

1. Center, Forward and Aft Forgings

These aluminum alloy sections, machined from 2014-T6 forgings, displayed a surface appearance similar to the adjacent tank shell, as previously described in Section V. A.

above. No unusual conditions were observed except for some minor end grain attack of the radiused area machined into the forward, oxidizer inlet, pressurizing passage component. This area exposed to the IRFNA oxidizer, is located immediately adjacent to the burst band weld joint and is shown in Figure 14.

2. Gas Generator Tube

The solid propellant gas generator assembly is positioned on the centerline of the thrust unit, within an aluminum cylindrical tube which forms the inner wall of the oxidizer tank. The O.D. IRFNA exposed surface of the gas generator tube was fairly clean, with a white to very faint green powdery deposit on the wrought metal and adjacent weld joint. This residue like deposit was easily removed, disclosing clusters of blotch like, shallow corrosion spots, identical in appearance to those observed on the tank shell surface. This anomaly was selected for the confirmatory analysis presented in Section V. (D). 2.

3. Thrust Chamber and Baffle

The thrust chamber is formed by the aluminum fuel tank inner wall and is composed of a tubular element positioned on the centerline of the fuel tank. The O.D. surface of the chamber is regeneratively cooled by propellant flowing through a corrugated aluminum baffle which envelopes the thrust chamber tube.

Both of these components were discolored to a gray-black hue, displaying the same appearance as the fuel tank shell. The surface film was tightly adherent, indicating a surface reaction with the MAF-1 fuel had probably occurred. Scraping a small area produced a white, flocculent substance, identical in appearance to the film observed on the fuel tank surface. The propellant exposed surface of the thrust chamber tube, which more closely resembled the appearance of the tank shell, was selected for the confirmatory analysis presented in Section V. (D) 3. Mechanical properties of the baffle element were determined and are included in Section VI.

4. Gas Diffusers

Gas diffusers are located in the oxidizer and fuel tanks, just aft of the aluminum burst bands, at the inlet ends of the tanks. The diffuser material was spectroscopically identified as Type 321 stainless steel. The fuel exposed diffuser was very clean, with no corrosion present. The oxidizer exposed diffuser presented an entirely different appearance, with an accumulation of the same reddish-brown deposits originally noted on the aluminum tank shell surface. This observation suggests the theory that these deposits are residues from the propellant, since they were found on aluminum surfaces as well. A general surface corrosion was noted in other, clean areas of the diffuser. A cross section through such an area is shown in the photomicrograph of Figure 4. This general corrosion, measuring approximately 1.5 mils deep, apparently resulted from exposure to the

IRFNA oxidizer during the 10-year storage period. A corrosion rate of this magnitude is not considered significant for material of 0.020 inch thickness.

5. Vortex Director Baffle

The vortex baffle is welded to the gas generator tube in the oxidizer tank and is positioned around the liquid propellant injection orifices. Only superficial, surface dulling of this component was observed. A loosely adhering, white to pale green, powdery deposit was present on the surfaces. This residue was easily removed, disclosing scattered, blotch-like, shallow corrosion spots identical in appearance to those observed on the gas generator tube and oxidizer tank shell surfaces.

6. Burst Bands

Pressurizing inlet passages to each propellant tank are sealed by welded aluminum burst bands which rupture under gas generator pressure during the initial phase of ignition. The burst bands are believed to be fabricated of a low alloy aluminum or possibly commercially pure 1100 aluminum.

The fuel burst band was in direct contrast to the other fuel exposed aluminum component surfaces, which had been discolored to a gray-black hue. Only a slight, dulling of the surface occurred, with negligible grain boundary pitting, barely visible at 60X magnification.

The oxidizer exposed burst band displayed a brighter metallic luster than the adjacent aluminum alloy surfaces.

However, the scattered blotch like corrosion patches observed on the tank shell and gas generator tube surfaces were also present on this component, indicating an identical corrosion mechanism. The depth of this area type corrosion was estimated to be comparable to that measured on the tank shell surface, i.e., 3 to 4 mils.

7. Shock Baffle

A conical shaped aluminum shock baffle is located in the oxidizer tank, formed to fit around the center forging pressurization passages. This thin, sheet metal component was dulled in appearance, with the same reddish-brown residues on the surface as were previously associated with the tank shell surface. The scattered surface corrosion occurring as a result of exposure to IRFNA for the 10-year storage period was similar to that observed on adjacent aluminum alloy components situated within the oxidizer tank.

8. Oxidizer Pressurizing Inlet Passage Assembly

This aluminum alloy component is welded to the gas generator tube at the aft end and to the oxidizer tank inlet forging at the forward end. The burst band surrounds this component and is also welded to it. Physical appearance of this component indicates that it was probably machined from a short section of heavy wall pipe.

The same general, area type corrosion previously observed on the tank shell surface also occurred on this component. Some minor, end grain attack was noted in a machined radius adjacent to the burst band weld. This condition is shown in Figure 5.

9. Shear Discs

The liquid propellant injection orifices are sealed by aluminum shear cups, welded into the central header, to prevent mixing of the hypergolic propellants prior to ignition. Negligible corrosion was observed on the fuel exposed shear discs. Only a finely divided, adherent deposit, yellowish-white in color was noted.

Shear disc surfaces exposed to the oxidizer propellant displayed a film of residual deposits, with patches of general surface layer corrosion observed beneath these deposits. Four of the discs are shown in Figure 6, with a magnified view of one disc shown in Figure 7. The appearance of the corrosion area is identical to that observed on the tank shell surface and on other aluminum alloy components exposed to the IRFNA oxidizer.

A section was taken through the corroded area of an oxidizer exposed shear disc, and a metallographic sample prepared. The photomicrograph of this area is shown in Figure 8. Metal attack initiates as extremely small shallow pits which join together, resulting in the area type corrosion observed.

10. Screens

The most pronounced corrosion observed occurred on the stainless steel screen material attached to the aluminum oxidizer shock baffle. Corrosion of the screen was quite extensive, as shown in the comparison photomacrographs of Figure 9. A cross section view is shown in Figure 10, together with an original, non-corroded section.

Corrosion observed on this screen element, situated in the oxidizer tank and assumed to be a Type 304 stainless steel composition, is considered normal. However, the nature of the corrosion process is very localized and the location of the corroded area coincides with water line markings on the tank shell. These observations would indicate that residual, diluted IRFNA oxidizer remaining after draining was probably responsible for the corrosion which occurred, rather than the long term propellant storage period.

The corroded wire diameter was measured as 0.004 inch, or 0.016 inch less than the original, nominal diameter of 0.020 inch. This correlates fairly closely with reported corrosion rates for Type 304 stainless steel in IRFNA of approximately .4 to .6 mils per year. No unusual corrosion mechanism was operative, only a general, dissolution of the austenitic stainless steel wire by the diluted IRFNA.

C. METALLURGICAL EXAMINATION OF WELDMENTS

In general, weldments exposed to the propellants were affected in the same manner as the 2014-T6 aluminum alloy tank shells. Negligible corrosion was observed. Only some minor, pitting corrosion of the retainer weld holding the oxidizer gas diffuser in place, and the gas generator tube to oxidizer pressurizing passage assembly weld, was observed. Contact between the retainer weld and stainless steel gas diffuser, which is cathodic with respect to aluminum, suggests the possibility of a galvanic corrosion mechanism as being at least partly responsible for the observed pitting. A photomacrograph of the most severely pitted area of the retainer weld is shown in Figure 11. A cross section of these pits, included in Figure 11, indicates the maximum depth of corrosion observed.

Some of the oxidizer exposed welds displayed the same scattered, layer type, area corrosion observed on the wrought aluminum surfaces. These blotch-like areas of corrosion were bright and shiny and were generally found beneath deposits of reddish-brown or yellowish-white propellant residues, which were easily flaked off to expose the shallow, corroded area. The degree of corrosion observed on the structural welds was considered to be insignificant. Cross sections of each weld examined and its description are presented in Figures 12 through 15.

D. METALLURGICAL ANALYSIS OF ANOMALIES

1. General

The specific anomalies selected for detailed and confirmatory analyses were based on the initial examinations performed on the thrust unit tankage and components, as described in previous sections.

A detailed and a confirmatory analysis for the oxidizer section and the fuel section of the missile thrust unit are required by the subject contract. These analyses are presented in this section.

2. Corrosion of Oxidizer Tank Shell Surface

(a) Test History

The oxidizer tank shell, an integral part of the Bullpup missile liquid propellant thrust unit body, is located in the forward section of the missile, in tandem with the fuel tank. It was loaded with inhibited red fuming nitric acid (IRFNA, Type III-A, per MIL-P-7254C) then placed in storage for a period of approximately 10 years. It was defueled for the primary purpose of recovering UDMH from the MAF-1 fuel. At the same time it afforded an excellent opportunity to evaluate the long term compatibility of the 2014-T6 aluminum alloy tank shell with the inhibited red fuming nitric acid (IRFNA) oxidizer.

(b) Observations

Scattered, rust colored deposits and considerable loose residue were present on the internal surface of the oxidizer tank. The major portion of the surface retained a dull, metallic luster, with scattered, light green, powdery deposits. Water line markings were also visible, indicating the possible evaporation of residual propellant or rinse fluids.

A two layer residue deposit, described in Section V. A. and shown in the photomacrograph of Figure 3, was removed from several areas for examination of the underlying metallic surface. Brightly etched clusters of shallow corroded areas were observed, as seen in the photomacrographs of Figures 16 and 17.

(c) Metallurgical Analysis

A cross section through one of the blotch-like areas of corrosion was mounted and prepared for metallographic examination, to verify the general, shallow, surface nature of the corrosion observed. The Figure 18 cross section view indicates the depth of corrosion occurring. Corrosion depth was quite shallow, measuring 3 to 4 mils in the areas of greatest penetration.

The blotch like corrosion pattern observed on the tank shell surface differs from the isolated, pitting type corrosion generally associated with aluminum alloys and observed on aluminum alloy tankage examined during the original contract effort (Reference 1). However, it does initiate as a cluster of extremely small, shallow pits which then expand in area rather than depth. This can be seen in the enlarged view of Figure 17. The pitting appears to be preferentially oriented, probably in the direction of grain flow.

A cross section view of the two layer residue deposit which covered the corroded areas is shown in Figure 19. The layer contiguous to the metal surface is dark green in color and the outer layer a tan color. The dark green layer was more adherent, less brittle and did not flake off like the outer layer. However, it was easily scraped off, indicating that it might possibly be a deposit of propellant or rinse solution residues. An analysis of these deposits and other loose residue found in the oxidizer shell disclosed the presence of fluorides and nitrates. The source of these fluorides could be HF acid, present in the IRFNA oxidizer as an inhibitor. This would indicate that some surface reaction with the aluminum tank had occurred. This is discussed further in Section V. F, "Residue and Corrosion Product Analysis".

(d) Confirmatory Analysis

The blotch like, area type corrosion discussed above was also observed on other aluminum alloy components contained within the tank shell and exposed to the IRFNA oxidizer. Visual examination of these components at magnifications to 60X clearly identified the mechanism of corrosion occurring as general surface corrosion, emanating from extremely small shallow pits and spreading in area rather than depth. A photomacrograph of a similarly corroded area occurring on the shock baffle surface is presented in Figure 20.

3. Corrosion of Fuel Tank Shell Surface

(a) Test History

The fuel tank shell, also an integral part of the Bullpup missile propellant thrust unit body, is located in the aft section of the missile, in tandem with the oxidizer tank. It was loaded with MAF-1 fuel (per RMD specification 4034) then placed in storage for a period of approximately 10 years. As with the oxidizer propellant tank shell, this long term storage afforded an excellent opportunity to evaluate the compatibility of 2014-T6 aluminum alloy with the mixed amine, MAF-1 fuel.

(b) Observations

The fuel tank surface was discolored to a gray-black color. Scraping a small area of the surface disclosed the presence of a tightly adhering film, indicating a surface reaction with the propellant had probably occurred. The scraped film was found to be a white, crystalline substance. A sufficient quantity was removed for X-ray diffraction and Infrared analysis. The surface beneath the film retained a metallic luster, with little evidence of pitting at magnifications to 60X.

(c) Metallurgical Analysis

Since no macroscopic evidence of corrosion was visible and an apparent surface reaction, similar to anodizing, had occurred, a metallographic cross section was prepared for high magnification examination of the metal surface. Photomicrographs of two exposures are shown in Figure 21. A very tight, adherent film is visible in the bottom exposure. Corrosion in the form of very small, shallow, scattered pits had occurred. Maximum pit depth was less than 1 mil. The degree of corrosion is considered negligible.

(d) Confirmatory Analysis

The gray-black discoloration observed on the fuel tank shell surface was also found on the components situated within the tank shell. The thrust chamber baffle, which had developed a surface finish and discoloration very similar to that of the tank shell, was selected for a confirmatory analysis. Visual examination of this component disclosed a very tightly adherent, continuous film, similar in appearance to an anodized film. Scraping a small area of this film produced a white, crystalline material identical to that observed on the tank shell surface, and indicative of a reaction product. The surface beneath the film retained a metallic luster, with very little evidence of pitting or general corrosion at magnifications to 60X.

A cross section of the chamber was cut and mounted for metallographic examination of the metal surface. The photomicrograph of this surface, shown in Figure 22, is very similar to the tank shell section shown in Figure 21 and also indicates a negligible degree of corrosion resulting from the long exposure to the MAF-1 fuel.

E. MECHANICAL PROPERTIES OF ALUMINUM ALLOY TANK SHELL, GAS GENERATOR TUBE AND THRUST CHAMBER BAFFLE MATERIAL

Three tensile test specimens were machined from each component, to verify the heat treat condition of the 2014-T6 aluminum alloy used in fabricating the tank shells and gas generator tube, and to determine if any deterioration of structure may have occurred over the 10-year storage period. The test results are summarized in Table I. Minimum values for 2014-T6 aluminum alloy sheet and pipe are included for comparison. These mechanical properties were easily met by the tank shell and gas generator tube material. This observation would indicate no effect on properties from the long term propellant exposure. Fracture characteristics of the specimens were normal, with no evidence of embrittlement or other corrosion related phenomena.

The thrust chamber baffle material, a 6061 aluminum alloy, displayed properties comparable to those expected for the -T4 condition, indicating no deleterious effects from propellant exposure. High ductility values recorded for this material in Table I are also an excellent indication of the compatibility of this alloy with the MAF-1 fuel. Fracture characteristics provided further evidence of a normal, ductile failure, with no evidence of embrittlement present.

F. RESIDUE AND CORROSION PRODUCT ANALYSIS

Residues were removed from the following areas:

1. Oxidizer Tank: IRFNA Oxidizer - loose. rust colored debris mixed with white, powdery deposits and a dark green film.

TABLE I

MECHANICAL PROPERTIES OF BULLPUP MISSILE LIQUID PROPELLANT THRUST UNIT TANK SHELLS AND SELECTED COMPONENTS

LOCATION OF TEST SPECIMENS	ALLOY IDENTIFICATION	SPECIMEN NUMBER	ULTIMATE TENSILE STRENGTH PSI	0.2% OFFSET YIELD STRENGTH PSI	% ELONGATION IN 2 INCHES
Oxidizer Tank Shell - 1/4 Inch Thick Wall	2014-T6	1	70,600	64,000	8*
		2	69,000	63,000	6*
		3	69,600	63,600	6*
		Average	69,700	63,500	7
Fuel Tank Shell - 1/4 Inch Thick Wall - Specimens Machined to 0.210 Inch	2014-T6	1	69,000	63,300	6*
		2	72,300	66,600	6*
		3	73,300	67,600	10
		Average	71,500	65,800	7
Gas Generator Tube From Oxidizer Tank	2014-T6	1	67,300	60,100	10
		2	66,800	60,100	10
		3	66,500	60,400	10
		Average	66,900	60,200	10
Thrust Chamber Baffle From Fuel Tank	6061-T4	1	39,800	24,200	25
		2	39,300	23,100	22
		3	39,800	23,700	25
		Average	39,600	23,700	24
Alcoa Minimum Properties	2014-T6	Sheet	66,000	58,000	7
		Pipe	60,000	53,000	7
	6061-T4	Sheet	30,000	16,000	16

* BROKE OUTSIDE OF GAGE MARKS

2. Gas Generator Tube: Exposed to IRFNA - scraped off whitish, powdery deposits.

3. Fuel Tank: MAF-1 Fuel - scraped off gray-black, adherent surface film and light green powder.

4. Thrust Chamber· MAF-1 Fuel - scraped off gray-black adherent surface film.

Infrared, X-ray diffraction and chemical spot test analyses were performed in an effort to identify the surface films and residue deposits removed from the exposed surfaces. The following corrosion products were identified:

COMPONENT	ANALYSIS TECHNIQUE		
	CHEMICAL SPOT TEST	INFRARED	X-RAY DIFFRACTION
Oxidizer Tank Shell and Gas Generator Tube	Fluorides	Hydrated Aluminum Nitrate Salts $AL(NO_3)_3$ 9H20	Aluminum Hydroxy Fluoride 16AlF $(OH)_2$ to 16AlF_2 (OH)
Fuel Tank Shell and Thrust Chamber		Aluminum Hydroxide	Beta Aluminum Hydroxide

These identified compounds are expected reaction products for the aluminum alloy/propellant combinations involved. No unusual aluminum compounds were formed. The total quantity of loose reaction products formed in the oxidizer tank shell was relatively small and should not interefere with the functional capability of the missile, even if these products were formed during the 10-year storage. However, it is not certain they were, since considerable time had elapsed between draining of the thrust unit and sectioning for the current investigation. Observed water line markings also indicate a strong possibility that post-storage reactions had occurred. These reactions would account for at least a portion of the residue present.

SECTION VI

OBSERVATIONS, CONCLUSIONS AND RECOMMENDATIONS

A. OBSERVATIONS AND CONCLUSIONS

The overall interpretation of the macroscopic and microscopic examinations performed during this program, and the detailed and confirmatory analyses of selected anomalies can be summarized briefly in the following observations and conclusions:

1. No cracking of welds or parent metal was noted.
2. No stress-corrosion effects, as a result of long term exposure to propellants, were noted.
3. No deleterious or pronounced corrosion effects were observed on either the primary tank shell surfaces or propellant exposed components.
4. Analysis of the residues removed from the oxidizer tank indicated only slight metal attack and dissolution. The major portion of the deposits was determined to be aluminum salts.
5. The tightly adherent film observed on the fuel tank interior and propellant exposed component surfaces were analyzed and found to be beta aluminum hydroxide.
6. Basic compatibility for periods of at least 10 years has been verified for the materials used in fabricating the Bullpup "B" missile liquid thrust unit with the propellants IRFNA (oxidizer) and MAF-1 (fuel).

B. RECOMMENDATIONS

Since corrosion of the thrust unit was almost negligible there is little basis for any extensive recommendations. In general, flushing and rinsing operations after removal of the propellant should be thorough with a final drying operation to remove all moisture. Enclosure in a sealed container, with a deliquescent compound such as calcium chloride included, should maintain propellant exposed surfaces of the unit in a stable, as exposed condition. Exclusion of moisture should prevent the occurrence of misleading, secondary reactions.

It might be desirable to consider changing the present stainless steel screen material in the oxidizer tank to an alloy composition more resistant to corrosion by the IRFNA propellant.

SECTION VII

REFERENCES

1. Technical Report AFRPL-TR-74-82 "Analysis of Liquid Rocket Tankage" Final Report, Bell Aerospace Company J. Salvaggi, H. G. Kammerer, E. J. King, April 1975.

2. Report RMD 2014-T1 "Qualification Test of YLR62-RM-2 Packaged Liquid Propellant Thrust Unit For The Bullpup "B" Missile" Thiokol Chemical Corporation, Reaction Motors Division, H. Fox, H. A. Jatczak, H. Davies, A. R. Maier, March 1962.

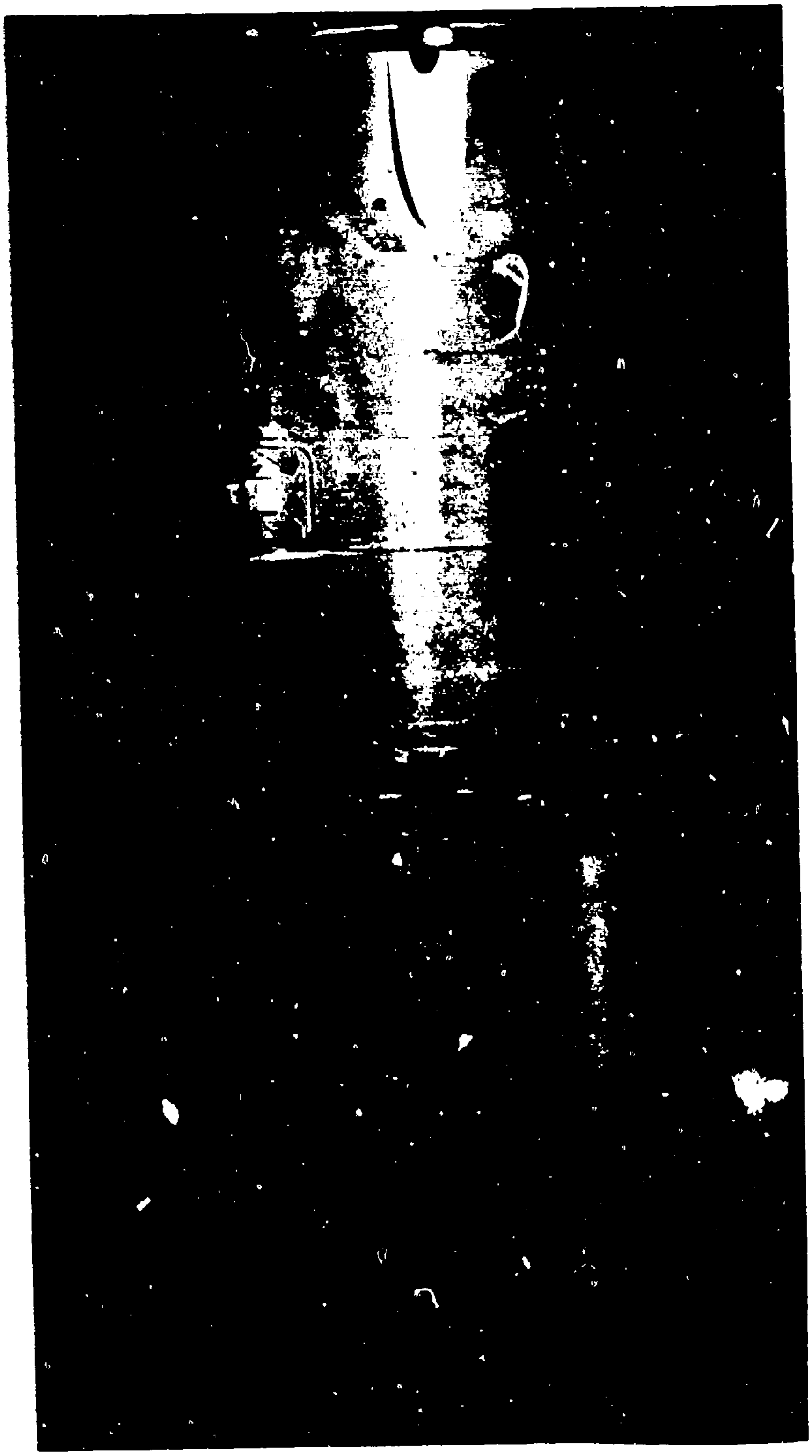


FIGURE 1. OVERALL VIEW OF BULLPUP MISSILE LIQUID PROPELLANT THRUST UNIT IN AS-RECEIVED CONDITION.

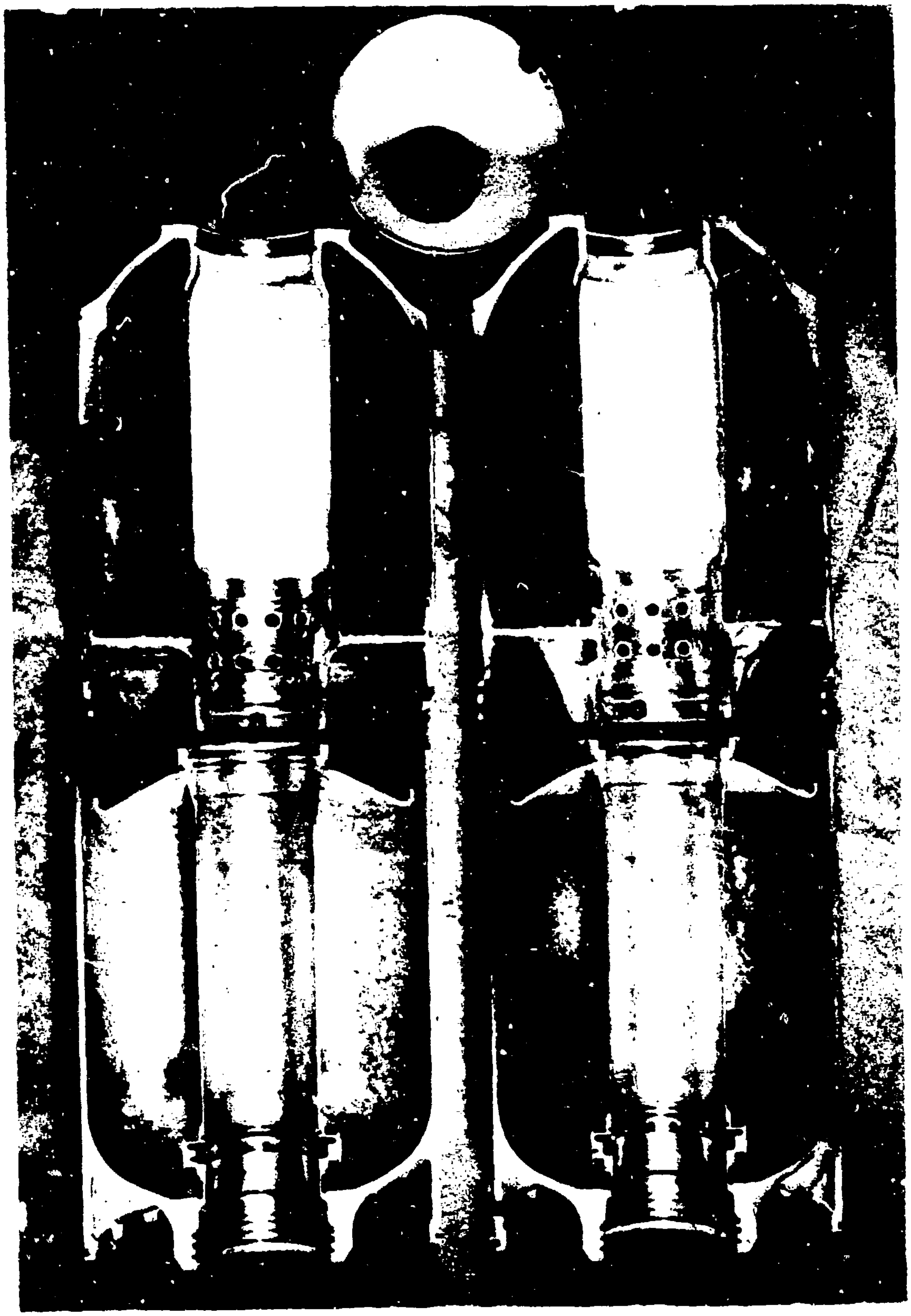
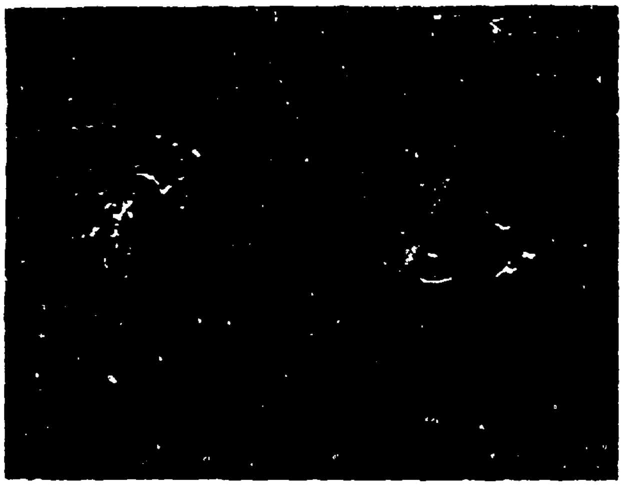


FIGURE 2. SECTIONED VIEW OF THRUST UNIT SHOWING INTERIOR CONDITION AFTER 10-YEAR STORAGE WITH PROPELLANTS.

MAG.: 6X



MAG.: 12X

FIGURE 3. TWO-LAYER SURFACE DEPOSITS OBSERVED ON INTERIOR OF OXIDIZER (IRFNA) TANK FROM BULL PUP MISSILE. SHINY AREA IS BARE METAL SURFACE WITH A SHALLOW LAYER REMOVED BY CORROSION.

FIGURE 4. GENERAL SURFACE CORROSION OF STAINLESS STEEL GAS DIFFUSER EXPOSED TO IRFNA, IN BULLPUP MISSILE OXIDIZER TANK, FOR TEN YEARS.

MAG.: 100X
ETCHANT: MIXED ACIDS

FIGURE 5. SHALLOW END GRAIN ATTACK OF ALUMINUM ALLOY, OXIDIZER INLET PRESSURIZING PASSAGE, ADJACENT TO BURST BAND WELD SHOWN IN FIGURE 14.

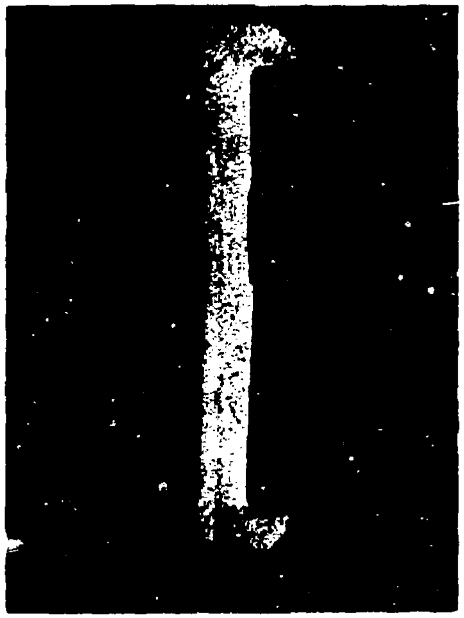
MAG.: 100X
ETCHANT: KELLER'S

FIGURE 6. PHOTOMACROGRAPH OF SHEAR DISCS REMOVED FROM OXIDIZER TANK, SHOWING RESIDUES ON SURFACE.

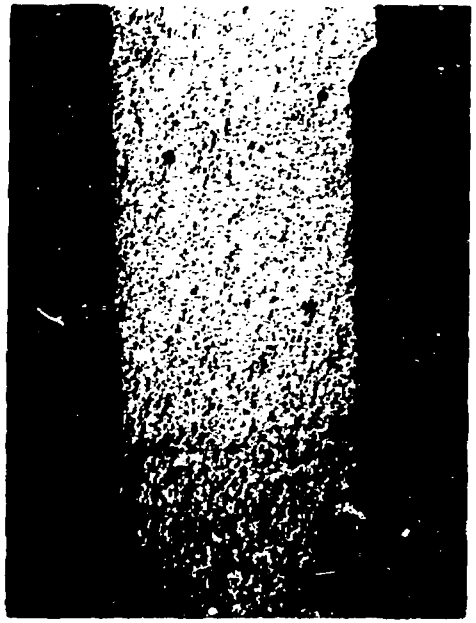
MAG.: 2-1/2X

FIGURE 7. MAGNIFIED VIEW OF ONE DISC IN FIGURE 6. SHOWING RESIDUE AND AREA OF GENERAL CORROSION.

MAG.: 6X



MAG.: 6X



MAG.: 32X

MAG.: 200X

FIGURE 8. CROSS SECTION THROUGH SHEAR DISC SHOWN IN FIGURE 7. SHOWING GENERAL CORROSION OCCURRING ON OXIDIZER EXPOSED SURFACE OF ALUMINUM ALLOY.

[redacted]

[redacted]

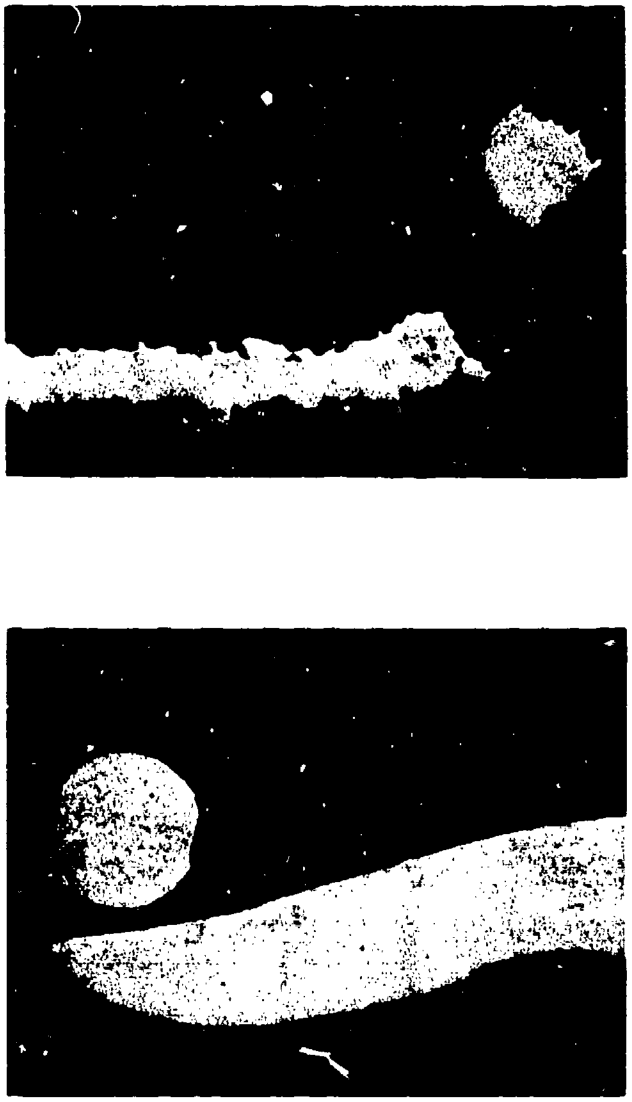
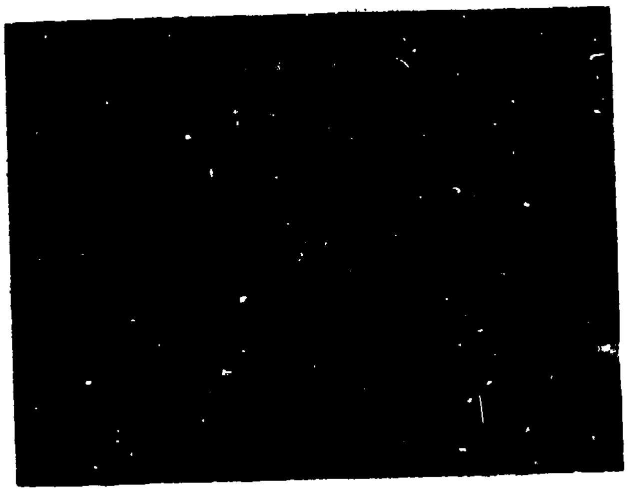
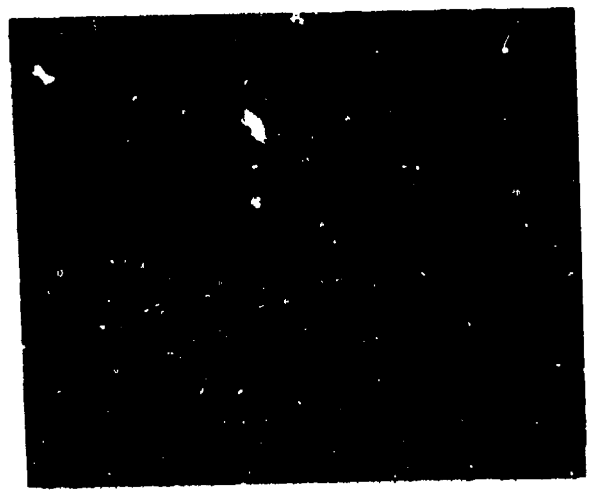


FIGURE 10. COMPARATIVE CROSS SECTIONS OF CORRODED AND NON-CORRODED SCREEN MATERIAL SHOWN IN FIGURE 9.

MAG.: 50X

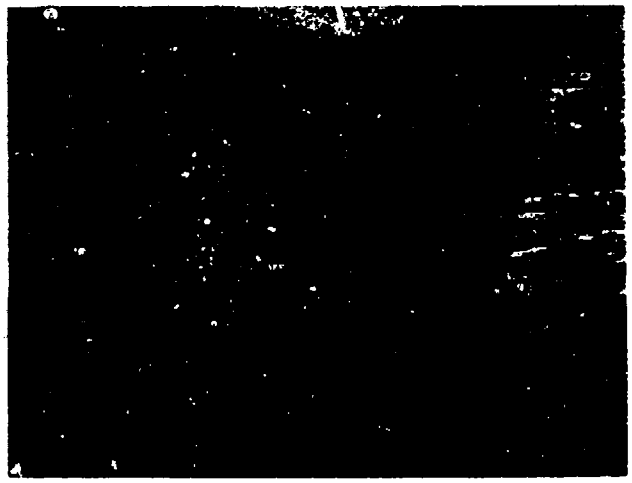


MAG.: 5X

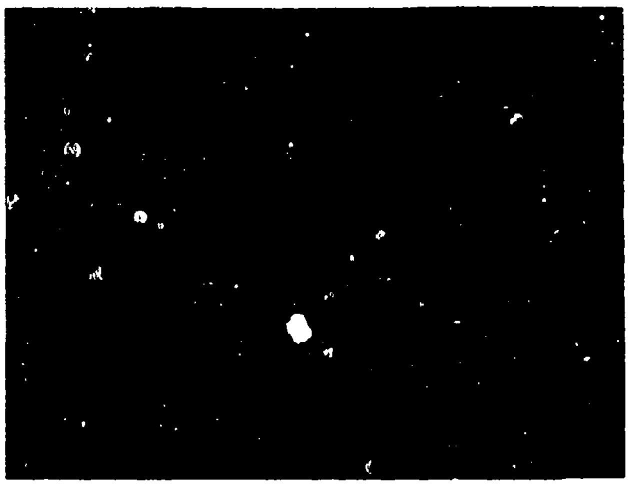


MAG.: 10X

FIGURE 11. PHOTOMACROGRAPHS OF ALUMINUM RETAINER WELD BEAD DEPOSITED ON CANISTER INLET BAFFLE. NOTE PITTED CONDITION OF WELD, BELIEVED DUE TO GALVANIC CORROSION RESULTING FROM CONTACT WITH STAINLESS STEEL INLET BAFFLE.



OXIDIZER SHELL WELD



FUEL SHELL WELD

FIGURE 12. SECTIONS THROUGH OXIDIZER AND FUEL TANK GIRTH WELDS, SHOWING CLEAN, NON-CORRODED STRUCTURES.

MAG.: 10X

FIGURE 13. SECTION THROUGH FUEL TANK BURST BAND WELD SHOWING CLEAN, NON-CORRODED STRUCTURE OF WELD AND PARENT MATERIAL.

MAG.: 10X

OXIDIZER [illegible]
[illegible]

GAS GENERATOR TUBE TO PRESSURE PASSAGE WELD AT OXIDIZER INLET END OF MISSILE - EXPOSED TO IRFNA

GAS GENERATOR TUBE TO OXIDIZER INLET FORGING - THIS WELD NOT EXPOSED TO IRFNA

FIGURE 14. SECTIONS THROUGH VARIOUS STRUCTURAL WELDS IN OXIDIZER TANK OF BULLPUP MISSILE. ALL MATERIALS ARE ALUMINUM ALLOYS.

MAG.: 6X
ETCHANT: KELLER'S

[redacted]

GAS GENERATOR TUBE TO VORTEX DIRECTOR BAFFLE - EXPOSED TO IRFNA

THRUST CHAMBER BAFFLE TUBE TO CENTRAL HEADER FORING - EXPOSED TO MAF-1 FUEL

FIGURE 15. SECTIONS THROUGH VARIOUS STRUCTURAL WELDS IN THE BULLPUP MISSILE. ALL MATERIALS ARE ALUMINUM ALLOY FORGINGS OR BAR STOCK.

MAG.: 6X
ETCHANT: KELLER'S

FIGURE 16. CLUSTER OF SHALLOW CORROSION SPOTS [illegible] BY REMOVAL OF SURFACE DEPOSITS ON [illegible] TANK SHELL.

[illegible]

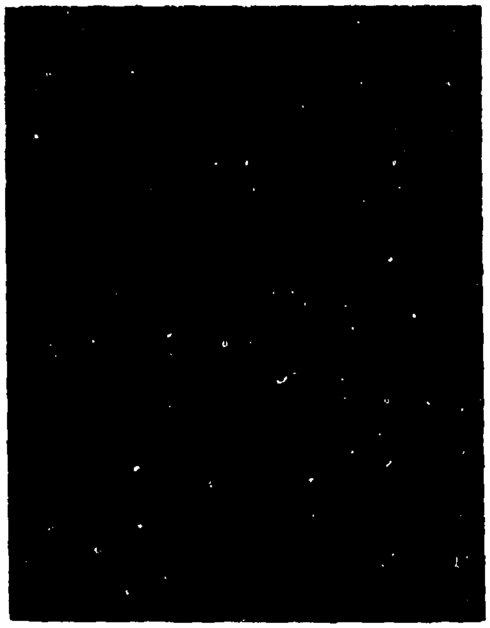


FIGURE 17. ENLARGED VIEW OF CORRODED AREA SHOWING VERY FINE PIT FORMATION WHICH PRECEDES THE REMOVAL OF A SURFACE LAYER OF METAL ON OXIDIZER TANK SHELL.

MAG.: 20X

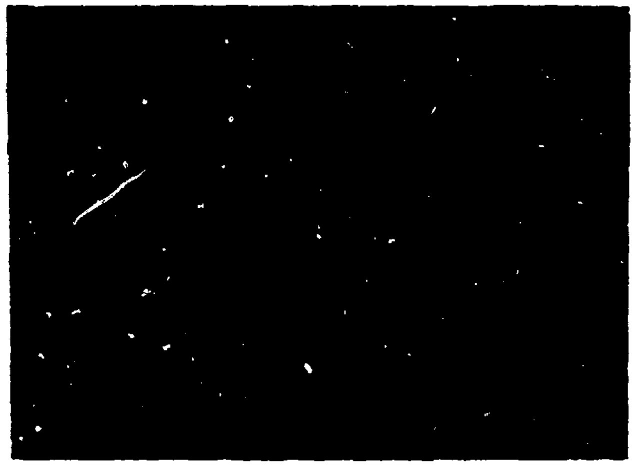


FIGURE 18. GENERAL SURFACE CORROSION OBSERVED ON 2014-T6 ALUMINUM ALLOY OXIDIZER TANK FROM BULLPUP MISSILE EXPOSED TO IRFNA FOR TEN YEARS

MAG.: 50X
UNETCHED

FIGURE 19. TWO-LAYER SURFACE DEPOSIT COVERING AREAS OF GENERAL CORROSION SHOWN IN FIGURE 16. FIRST LAYER IS DARK GREEN IN COLOR. OUTER LAYER IS LIGHT TAN AND WAS EASILY FLAKED OFF.

MAG.: 50X
UNETCHED

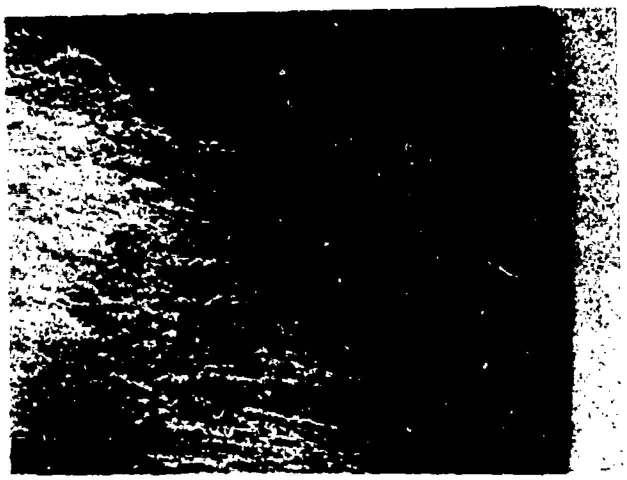


FIGURE 20. PHOTOMACROGRAPH OF AREA-TYPE CORROSION OCCURRING ON OXIDIZER EXPOSED SHOCK BAFFLE. IDENTICAL TO THAT OBSERVED ON THE OXIDIZER TANK SHELL, AS SHOWN IN FIGURE 16.

MAG.: 6X

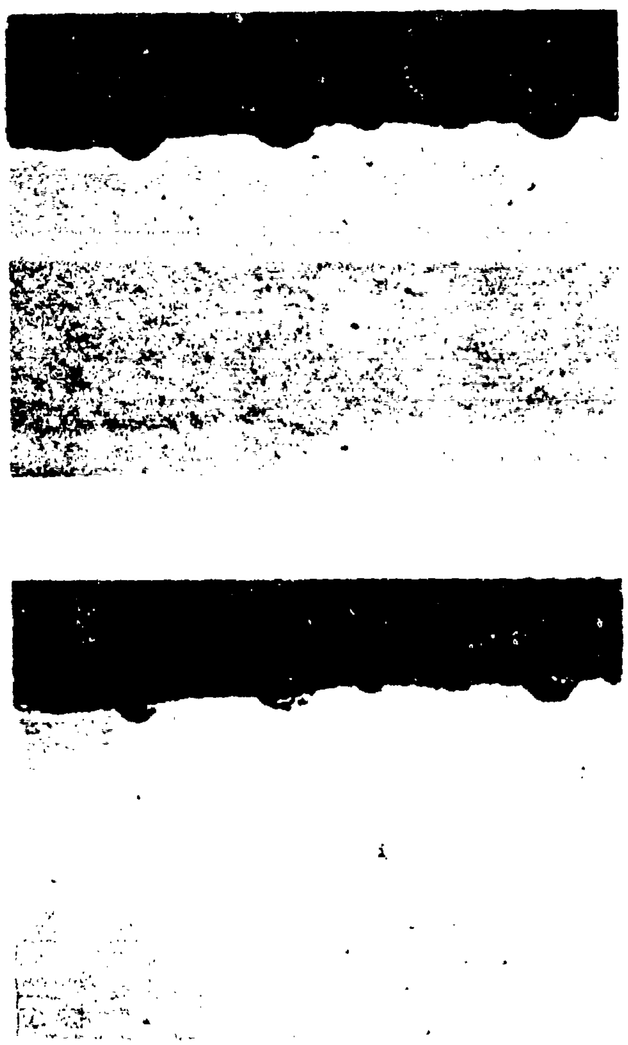


FIGURE 21. VERY SHALLOW PITTING CORROSION OBSERVED ON 2014-T6 ALUMINUM ALLOY FUEL TANK FROM BULLPUP MISSILE, EXPOSED TO MAF-1 FUEL FOR TEN YEARS. CORROSION LAYER IN BOTTOM VIEW IS DARK GRAY AS OBSERVED VISUALLY.

MAG.: 200X
UNETCHED

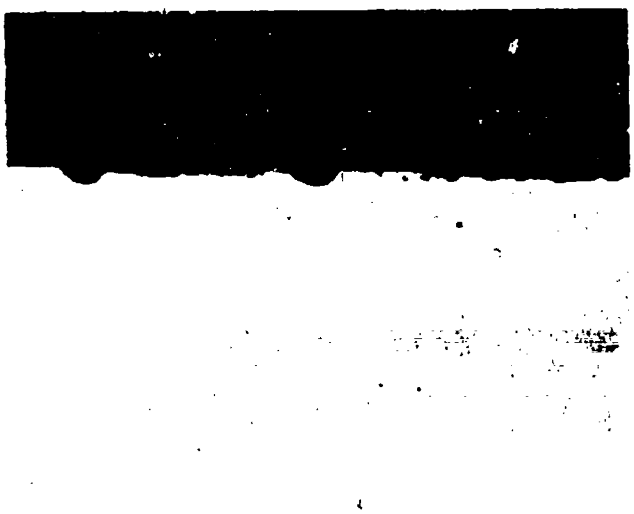


FIGURE 22. PHOTOMICROGRAPH OF FUEL EXPOSED THRUST CHAMBER CROSS SECTION, WITH PITTING CORROSION AND SURFACE FILM IDENTICAL TO THAT OBSERVED ON FUEL TANK SHELL SURFACES AS SHOWN IN FIGURE 21.

MAG.: 200X
UNETCHED